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Rapid separation method for emergency water and urine samples

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The Savannah River Site Environmental Bioassay Lab participated in the 2008 NRIP Emergency Response program administered by the National Institute for Standards and Technology (NIST) in May, 2008. A new rapid column separation method was used for analysis of actinides and ^{90}Sr in the NRIP 2008 emergency water and urine samples. Significant method improvements were applied to reduce analytical times. As a result, much faster analysis times were achieved, less than 3 hours for determination of ^{90}Sr and 3–4 hours for actinides. This represents a 25%–33% improvement in analysis times from NRIP 2007 and a ~100% improvement compared to NRIP 2006 report times. Column flow rates were increased by a factor of two, with no significant adverse impact on the method performance. Larger sample aliquots, shorter count times, faster cerium fluoride microprecipitation and streamlined calcium phosphate precipitation were also employed. Based on initial feedback from NIST, the SRS Environmental Bioassay Lab had the most rapid analysis times for actinides and ^{90}Sr analyses for NRIP 2008 emergency urine samples. High levels of potential matrix interferences may be present in emergency samples and rugged methods are essential. Extremely high levels of ^{210}Po were found to have an adverse effect on the uranium results for the NRIP-08 urine samples, while uranium results for NRIP-08 water samples were not affected. This problem, which was not observed for NRIP-06 or NRIP-07 urine samples, was resolved by using an enhanced ^{210}Po removal step, which will be described.

Introduction

There is an increasing need to develop faster analytical methods for emergency response, including emergency water and urine samples.[1–3] Actinide methods using alpha-spectrometry referenced in the literature typically refer to these methods as having tedious sample preparation techniques and long counting times, inappropriate for rapid analysis.[4,5] The SRS Environmental Bioassay Lab participated in the NRIP 2007 Emergency Response Program administered by the National Institute for Standards and Technology (NIST) in May, 2007, reporting actinides and ^{90}Sr in record times,[6] indicating this description is no longer accurate for all alpha-spectrometry methods. The SRS technology also allows the determination of actinides with relatively short half-lives, such as ^{238}Pu, ^{241}Am, and ^{244}Cm, which are very difficult to determine by inductively coupled plasma mass spectrometry (ICP-MS) due to the relatively low mass of these isotopes. To determine if the analytical times for these rapid analyses could be reduced even further, additional improvements were developed. These improvements were tested when the Savannah River Site (SRS) Environmental Bioassay Lab participated in the 2008 NRIP Emergency Response Program in May, 2008.

A more rapid separation method was applied to the NRIP 2008 emergency water and urine samples, with streamlined sample preparation to reduce preparation time. Calcium phosphate precipitation, used previously to pre-concentrate actinides and ^{90}Sr in NRIP urine and water samples, was streamlined for faster processing of the NRIP 2008 urine and water samples. Column flow rates were doubled, with significant time savings realized. In addition, larger sample aliquots were taken, and as a result, count times were cut in half without sacrificing analytical quality. Emergency samples may contain much higher levels of interferences than are typically seen in routine samples, so emergency sample methods must be rugged. This was observed for uranium results on some of the NRIP 2008 urine samples. Results and subsequent testing to identify this interference will be described, along with alternate options to improve the ruggedness of the method.

Experimental

Reagents

The resins employed in this work are TEVA Resin® (Aliquat ™336), TRU-Resin® (tri-n-butylphosphate (TBP) and N,N-diisobutylcarbamoylmethylphosphine oxide (CMPO)), and Sr-Resin® (4,4',(5') di-t-butylcyclohexane-18-crown-6), available from Eichrom Technologies, Inc., (Darien, Illinois, USA). Nitric, hydrochloric and hydrofluoric acids were prepared from reagent-grade acids (Fisher Scientific, Inc.). All water was obtained from a Milli-Q2™ water purification system. All other materials were ACS reagent grade. Radiochemical isotope tracers ^{242}Pu, ^{243}Am, and ^{232}U that were obtained from Analytics, Inc. (Atlanta, GA, USA) and diluted to the approximately 0.37 Bq·mL^{-1} level were employed to enable yield corrections. ^{232}U tracer was prepared to be self-cleaning, removing its ^{228}Th daughter using barium sulfate precipitation.[7] ^{90}Sr standardized solution was obtained from Analytics, Inc. (Atlanta, GA, USA) and diluted to approximately 2.96 Bq·mL^{-1}.

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A solution of 20.95 mg·mL^{-1} stable strontium was used to determine strontium carrier recovery. The strontium carrier solution was standardized gravimetrically using a strontium carbonate precipitation technique. An aliquot containing 4.19 mg of Sr carrier solution (equivalent to 10.12 mg $Sr(NO_3)_2$ when evaporated on a planchet) was added to each sample.

Procedures

Column preparation: TEVA, TRU, and Sr-Resin columns were obtained as cartridges containing 2 mL of each resin from Eichrom Technologies, Inc. Small particle size (50–100 μm) resin was employed, along with a vacuum extraction system (Eichrom Technologies). Flow rates of 1–2 mL·min^{-1} have typically been used in the SRS Environmental Bioassay Laboratory, but flow rates were increased by factor of two for this work.

Sample preparation: After urine and water sample aliquots were dispensed (100 mL-urine; 400 mL-water), 2 mL 1.25M calcium nitrate (100 mg Ca) and 5 mL 3.2M ammonium hydrogen phosphate were added to each sample. For samples, the sample dispensing and the above reagent additions were performed in 225 mL (urine) or 500 mL (water) centrifuge tubes to save time. The pH was adjusted to pH 10 with concentrated ammonium hydroxide using a dark pink phenolphthalein endpoint. For dark urine samples, pH paper may be used. Previously, after discarding the supernatant, the precipitate was rinsed once with 10–15 mL of water and centrifuged at 3000 rpm for ~5 minutes. For NRIP-08 samples, the water rinse was not performed to save time. For water samples, the precipitate was dissolved in 8 mL 6M HNO_3 and 8 mL 2M $Al(NO_3)_3$ directly in the centrifuge tubes. The final load solution contains 16 mL 3M HNO_3 and 1M $Al(NO_3)_3$. The aluminum nitrate was previously scrubbed to remove trace uranium by passing approximately 250 mL 2M aluminum nitrate through a large column (Environmental Express, Mount Pleasant, SC, USA) containing 7 mL of UTEVA Resin® (Eichrom Technologies) at ~10 mL/min. The column was prepared from a water slurry of the UTEVA resin. Previously, for NRIP-06 urine samples, the calcium phosphate precipitate was transferred to a 250-mL glass beaker using a small volume of concentrated nitric acid, evaporated and wet-ashed to help destroy residual organics from the urine. For NRIP-08 urine samples, no wet-ashing was performed on the urine samples to save time, saving 30–45 minutes in sample preparation time.

Column separation: The following column separation was performed, as outlined in a previous paper.[8] TEVA, TRU, and Sr-Resin cartridges were stacked on the vacuum box from top to bottom, in that order. Fifty milliliter centrifuge tubes were used to collect rinse or final purified fractions. Flow rates were increased significantly. Column load solutions were loaded at ~2 drops per second, rinse solutions at ~3–4 drops per second and column strip solutions were added at ~2 drops per second using vacuum.

A valence adjustment was performed by adding 0.5 mL 1.5M sulfamic acid and 1.25 mL 1.5M ascorbic acid with a three minute wait step to reduce plutonium to Pu^{3+} followed by 2 mL 3.5M sodium nitrite to oxidize plutonium to Pu^{4+}. After the valence adjustment, the sample solution was loaded onto the stacked column at approximately ~2 drops per second. After the sample was loaded, a tube rinse of ~3 mL 3M HNO_3 was transferred to the stacked column and a rinse of 5 mL 3M HNO_3 was added directly to the stacked column. The TRU Resin and Sr-Resin cartridges were removed and the TEVA cartridges were kept on the vacuum box. The TEVA cartridge was rinsed with 15 mL 3M nitric acid to remove sample matrix components. To elute thorium from TEVA Resin, 20 mL 9M hydrochloric acid were added and discarded.

The plutonium was stripped from TEVA Resin with 20 mL 0.1M hydrochloric acid–0.05M hydrofluoric acid –0.03M titanium(III) chloride**.** Fifty micrograms of cerium as cerium nitrate were added to the tubes, along with 1 mL of concentrated hydrofluoric acid (49%), prior to elution of the plutonium to reduce microprecipitation wait times. A 0.5 mL volume of 30 wt% hydrogen peroxide was added after the plutonium was eluted to oxidize any residual uranium to U^{6+} as a precaution. After waiting 10 minutes, the solutions were filtered onto 0.1 μm 25 mm polypropylene filters (Resolve® filter, Eichrom Technologies) and counted by alpha-spectrometry.

The TRU cartridges were placed on a separate vacuum box and processed at the same time as the TEVA Resin cartridges to save time. Americium was stripped from TRU Resin with 15 mL 4M HCl at ~2 drops per second. Cerium was added as described previously to the tubes, along with 3 mL of concentrated hydrofluoric acid (49%), prior to elution to reduce microprecipitation wait times. This solution was diluted to a total volume of 30 mL with water to reduce the acidity. After waiting 10 minutes, the solutions were filtered onto 0.1 μm 25 mm polypropylene filters and counted by alpha-spectrometry.

TRU Resin was rinsed with 12 mL 4M HCl–0.2M HF at ~2–3 drops per second to remove any residual thorium that may have passed through the TEVA cartridge and was retained on TRU Resin. Uranium was stripped from TRU Resin using 15 mL 0.1M ammonium bioxalate at ~2 drops per second**.** Cerium was added as described previously to the tubes, along with 1 mL of concentrated hydrofluoric acid (49%), prior to elution to reduce microprecipitation wait times. A 0.5 mL volume of 20 wt% titanium chloride was also added to each tube also prior to elution to reduce uranium to U^{4+}. After

waiting 10 minutes, the solutions were filtered onto 0.1 µm 25 mm polypropylene filters and counted by alpha-spectrometry.

The Sr-Resin cartridges were placed on a vacuum box and rinsed with 15 mL 8M HNO_3 at ~2–3 drops per second. The ^{90}Sr was stripped from the Sr-Resin using 10 mL 0.05M HNO_3 into 50-mL tubes at ~2 drops per second. This solution was transferred to preweighed planchets and evaporated on a hot plate to dryness. A 3 mL volume of 0.05M HNO_3 was used to rinse each tube and then was transferred to each planchet and dried. The dried planchets were allowed to cool and then were weighed to determine gravimetric carrier recovery**.** The planchets were counted by gas proportional counting.

Actinide filters were counted by alpha-spectrometry for approximately 30 minutes for urine and 45 minutes for water samples. Strontium count times using gas proportional counting were ten minutes.

Apparatus

Plutonium, americium, and uranium measurements were performed by alpha-particle pulse-height measurements using Passivated Implanted Planar Silicon (PIPS) detectors. ^{90}Sr measurements were performed using a Tennelec LB 4100 gas proportional counter. Polycarbonate vacuum boxes with 24 positions and a rack to hold 50 mL plastic tubes were used. Two boxes were connected to a single vacuum source by using a T-connector and individual valves on the tubing to each box.

Results and discussion

Table 1 shows the improvement in turnaround times for actinides and ^{90}Sr in NRIP 2008 urine and water samples compared to NRIP 2006 and NRIP 2007 turnaround times. The SRS Environmental Bioassay Lab reported ^{90}Sr, $^{239/240}$ Pu, ^{238}Pu, ^{234}U, ^{235}U, ^{238}U, and ^{241}Am in urine and water samples well within the 8 hour target time. ^{90}Sr in NRIP urine samples was reported in only 2.9 hours, a significant improvement over the 3.9 hour time in 2007. Actinide isotopes were reported in 3.1 to 4.2 hours, faster than the 4.6 to 5.2 hour times in 2007. ^{90}Sr in NRIP water samples was reported in only 3.2 hours, a significant improvement over the 4.25 hour time in 2007. Actinide isotopes were reported in 3.5 to 4.6 hours, faster than the 4.9 to 5.6 hour times in 2007.

If $^{89/90}Sr$ differentiation is needed, there are Čerenkov counting techniques for more rapid determination of ^{89}Sr and ^{90}Sr. ^{89}Sr can be measured directly by Čerenkov counting, employing methodology that takes advantage of the high Čerenkov counting efficiency of ^{89}Sr relative to ^{90}Sr.[9,10]

Table 2 shows the average difference of the SRS measured values for NRIP-2008 water and urine samples versus the NIST reference values. The average difference from NIST reference values for the average results from five samples ($N=5$) containing approximately three different levels of activity is shown for each analyte. Considering the short count time of ~45 minutes, the accuracy of the average measured values ($N=5$) for water samples was good, more than adequate for emergency response screening. The same samples were also recounted later to determine the effect of a longer count time. The recounted values for NRIP-08 urine samples were similar, with no improvement observed in the slight positive bias for the plutonium results. The recounted samples showed a slight reduction in bias for the determination of ^{241}Am. The slight positive bias for the plutonium results in urine was not observed for the plutonium in NRIP-08 water sample results, even though the methods were essentially identical. This slight bias for plutonium would be acceptable, however, for emergency response screening the differences are within the ~±30% uncertainties reported for these results. The uranium bias, which was traced to high ^{210}Po levels, will be discussed further when individual uranium results are presented below.

Table 1. Improved turnaround times (in hours) on NRIP-08 urine and water samples

Nuclide	Sample		
	NRIP 2006	NRIP 2007	NRIP 2008
Urine			
^{241}Am	7.4	4.6	3.1
$^{238/239}Pu$	7.4	4.8	3.3
$^{234,235.238}U$	7.4	5.2	4.2
^{90}Sr	5.8	3.9	2.9
Water			
^{241}Am	7.2	4.9	3.5
$^{238/239}Pu$	7.2	5.5	3.9
$^{234,235.238}U$	7.2	5.6	4.1
^{90}Sr	4.6	4.25	3.2

Table 2. NRIP-2008 water and urine analysis average results

Nuclide	Avg. Difference Reported vs NIST	Avg. Difference Longer Recounts
Water		
^{238}Pu	13%	6.3%
^{240}Pu	–2.3%	–4.5%
^{241}Am	9.6%	1%
^{238}U	–0.5%	–5.4%
^{234}U	9.0%	–6.7%
^{90}Sr	–14%	N/A
Urine		
^{238}Pu	24%	24%
^{240}Pu	16%	18%
^{241}Am	6%	1%
^{238}U	–41%	–1.6%*
^{234}U	–46%	–3.1%*
^{90}Sr	1.7%	N/A

Actinides: 30–45 minute count time/recounts: 2 hour count time.
*With additional purification.

Table 3 shows the SRS reported values compared with the NIST reference values for ^{241}Am in water for each sample analyzed. The differences, which range from –9% to +19%, fall within the reported uncertainty ranges for each reported result at the 95% confidence level. Table 3 also shows the SRS values when the same samples were recounted for 2 hours. The differences from the NIST values range from 0% to +13%, reducing the biases slightly. The ^{241}Am results, reported in only 3.5 hours, was excellent with a bias of <10%.

Table 4 shows the SRS reported values compared with the NIST reference values for ^{241}Am in urine for each sample analyzed. The differences, which range from –3% to +12%, fall within the reported uncertainty ranges for each reported result at the 95% confidence level. The ^{241}Am results, reported in only 3.1 hours, were excellent with an average bias of only 6%. Table 4 also show the SRS values when the same samples were recounted for 2 hours. The differences from the NIST values range from –0.8% to +7%, reducing the average bias to only 1%.

Table 3. NRIP-2008 water analysis results for ^{241}Am

Sample ID	NIST value, Bq·Smp^{-1}	SRS reported value Bq·Smp^{-1}±%, *k*=2	Difference, ±%
9	0.765	0.871±21%	+14
13	0.649	0.587±21%	–9
16	0.445	0.491±23%	+10
27	0.445	0.530±22%	+19
42	0.175	0.199±30%	+14
			Avg. +9.6%
Sample ID	NIST value, Bq·Smp^{-1}	SRS 2-hr count, Bq·Smp^{-1}±%, *k*=2	Difference, ±%
9	0.765	0.799±17%	+4
13	0.649	0.673±17%	+4
16	0.445	0.503±19%	+13
27	0.445	0.445±19%	0
42	0.175	0.188±23%	+7
			Avg. +5.8%

Table 4. NRIP-2008 urine analysis results for ^{241}Am

Sample ID	NIST value, Bq·Smp^{-1}	SRS reported value Bq·Smp^{-1}±%, *k*=2	Difference, ±%
724	0.1891	0.203±31%	+7
727	0.1965	0.221±29%	+12
735	0.4226	0.456±26%	+8
736	0.3759	0.366±27%	–3
742	0.4675	0.499±25%	+7
			Avg. +6%
Sample ID	NIST value, Bq·Smp^{-1}	SRS 2-hr count, Bq·Smp^{-1}±%, *k*=2	Difference, ±%
724	0.1891	0.195±19%	+3
727	0.1965	0.197±19%	+0.3
735	0.4226	0.409±16%	–3
736	0.3759	0.401±16%	+7
742	0.4675	0.464±16%	–0.8
			Avg. +1%

Table 5. 210Po to 232U tracer ratios for NRIP-08 urine and water samples

Sample ID	^{210}Po added, Bq	^{210}Po/^{232}U, ratio	Bias, %
NRIP-08 urine samples			
724	0.385	1.05	–25
727	0.400	1.09	–13
735	0.860	2.35	–63
736	0.764	2.09	–49
742	0.951	2.60	–55
NRIP-08 water samples			
9	0.622	1.70	13.6
13	0.528	1.44	–0.2
16	0.362	0.99	–4.8
27	0.362	0.99	–0.8
42	0.142	0.39	–10.4

Table 5 shows the SRS reported values compared with the NIST reference values for ^{238}U for each urine sample analyzed. The negative biases for samples 735, 736 and 742 urine samples are much greater than the biases of samples 724 and 727. The ^{210}Po levels were examined relative to the ^{232}U tracer level added to each sample. The larger negative biases (–49% to –63%) correlate with the higher ^{210}Po/^{232}U ratios. The water sample results, which had lower ^{210}Po/^{232}U ratios, did not show any significant negative bias. ^{210}Po (5.30 MeV) has an unresolvable alpha energy from ^{232}U (5.26, 5.32 MeV), so ^{210}Po must be effectively removed to avoid an adverse impact on the chemical yield when ^{232}U is used as a tracer.

To determine if ^{210}Po in the uranium fraction was responsible for the negative bias for the NRIP-08 urine samples, an effort was made to redissolve and repurify these samples. The cerium fluoride filters for the NRIP-08 urine sample were treated with 3M HNO_3–0.25M H_3BO_3 and warmed on a hot plate to redissolve the samples. The redissolved samples were loaded onto TRU Resin, which had been preconditioned with 5 mL 3M HNO_3. After loading the samples, the TRU Resin was rinsed with 15 mL 8M HNO_3 at 1–2 drops per second to remove any ^{210}Po present, and then uranium was eluted with 15 mL 0.1M ammonium bioxalate at ~1–2 drops per second. The remainder of the procedure continued as previously noted.

Table 6 shows the results after the redissolution and enhanced ^{210}Po removal. The average bias was –1.6% with a range of –5.2% to +2.0%. Two additional NRIP urine samples were obtained from NIST and were analyzed, this time adding enhanced ^{210}Po removal steps. For the first sample, TRU Resin was rinsed with 15 mL 8M HNO_3 at 1–2 drops per second just prior to rinsing with 4M HCl–0.2M HF, as described previously for the initial set of NRIP-08 urine samples.

Table 6. NRIP-2008 urine results for ^{238}U after enhanced ^{210}Po removal

Sample ID	NIST value, Bq·Smp^{-1}	SRS reported value, Bq·Smp^{-1}	Difference, ±%
724	0.2137	0.223	+4.4
727	0.2220	0.209	–5.9
735	0.4776	0.487	+2.0
736	0.4248	0.412	–3.0
742	0.5284	0.501	–5.2
			Avg –1.6
729*	0.228	0.217	–4.8
730**	0.181	0.181	0

Analysis of additional NRIP samples using enhanced ^{210}Po removal options.
* 15 mL 8M HNO_3 rinse-TRU Resin.
** Added reductant (15 mL 4M HCl–0.2M HF–0.001M $TiCl_3$)-TRU Resin (no 8M HNO_3).

The second sample was rinsed with 15 mL of 4M HCl–0.2M HF–0.001M $TiCl_3$ instead of 4M HCl–0.2M HF, with no 8M HNO_3 rinse added, to reduce Po^{4+} to unretained Po^{2+}. The ^{238}U results showed a bias of only –4.8% for the first sample and 0% bias for the second sample. The enhanced ^{210}Po removal steps effectively removed ^{210}Po in both cases. This demonstrates the negative bias was caused by the high levels of ^{210}Po present in that sample, and points out the need for rigorous separation techniques, especially for emergency samples. The additional ^{210}Po removal with 15 mL with 8M HNO_3 took only about 10 minutes longer to complete. Since the 4M HCl–0.2M HF column rinse was performed anyway as previously noted, adding the titanium chloride reductant did not increase the separation time at all.

Figure 1 shows an example of the plutonium spectra for the NRIP 2008 water samples. The ^{242}Pu tracer recovery was 99.8% and the full width at half maximum (FWHM) was 46.7 keV, showing acceptable alpha-peak resolution and minimal reduction in tracer recoveries even with much faster column flow rates. The ^{239}Pu peak labeled on the spectra represents ^{239}Pu plus ^{240}Pu, since these isotopes have essentially the same alpha energy.

Figure 2 shows an example of the plutonium spectra for the NRIP 2008 urine samples. The ^{242}Pu tracer recovery was 98.5% and the full width at half maximum (FWHM) was 25.8 keV, showing acceptable alpha-peak resolution.

The minimum detectable activity (MDA) for the actinides is calculated according to the following equation:[11]

$$MDA = [3+4.65\sqrt{B}]/(CT \cdot R \cdot V \cdot Eff \cdot 0.060)$$

where B is the total background counts = BKG (rate)·BKG count time, CT is the sample count time (min), R is the chemical recovery, V is the sample volume (liters), Eff is the detector efficiency, 0.060 is the conversion from dpm to mBq.

In low-level counting, where a zero background count is quite common, the constant 3 is used to prevent an excessively high false positive rate.

Figure 3 shows the minimum detectable activity using the method for 100 mL and 400 mL sample aliquots versus count time for actinides. The MDA can be adjusted as needed, depending on the sample aliquot and count time. For a 100 mL sample aliquot, the MDA for a 2-hour count time is 16.3 mBq·L^{-1}. For a 400 mL sample aliquot and 2-hour count time the MDA is 4.07 mBq·L^{-1}. For a 100 mL sample aliquot, the MDA for a 22-hour count time is 1.5 mBq·L^{-1}. For a 400 mL sample aliquot and 22-hour count time the MDA is 0.37 mBq·L^{-1}. The MDA for ^{90}Sr is calculated in a similar fashion and was determined to be equal to 260 mBq·L^{-1} (7.22 pCi·L^{-1}) for a 400 mL water sample counted for 10 minutes.

For emergency response screening, the SRS NRIP 2008 urine and water data quality is sufficient, but if improved accuracy, precision or lower MDA were needed the samples could have been counted longer. The column chemistry is rapid and flexible, and has been applied to other sample types such as digested air filters or vegetation once the samples are dissolved into the column load solution.[12]

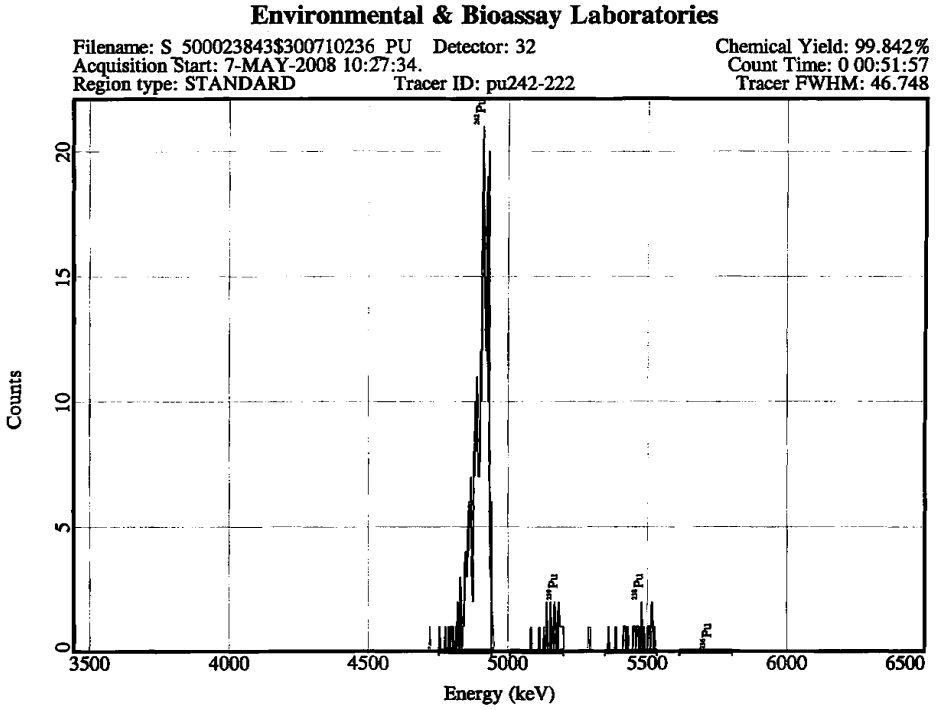

Fig. 1. Alpha-spectra showing Pu isotopes in NRIP 2008 water samples

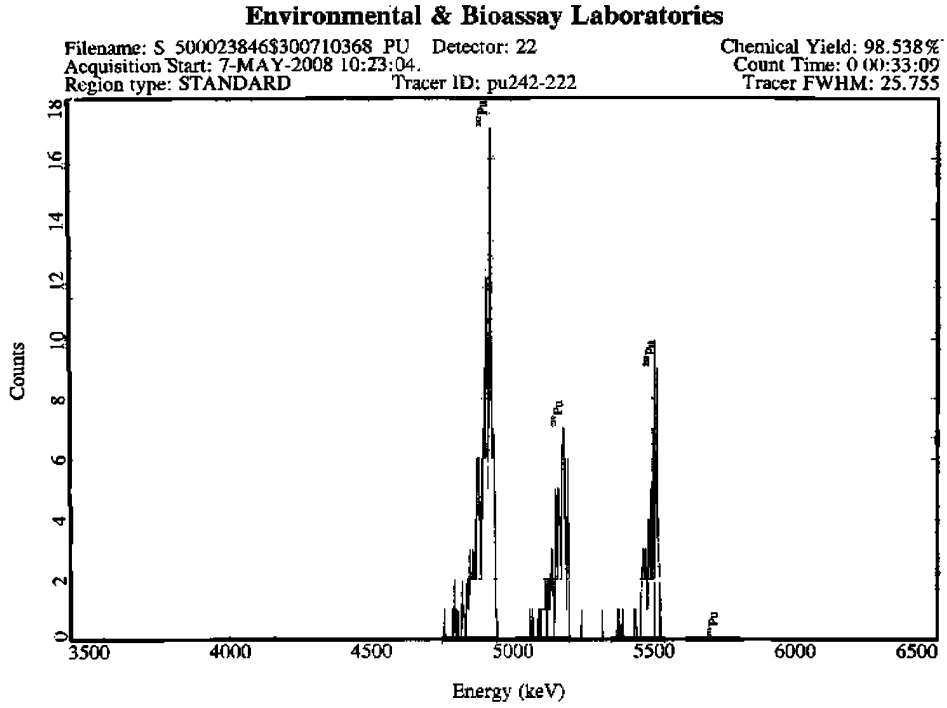

Fig. 2. Alpha-spectra showing Pu isotopes in NRIP 2008 urine samples

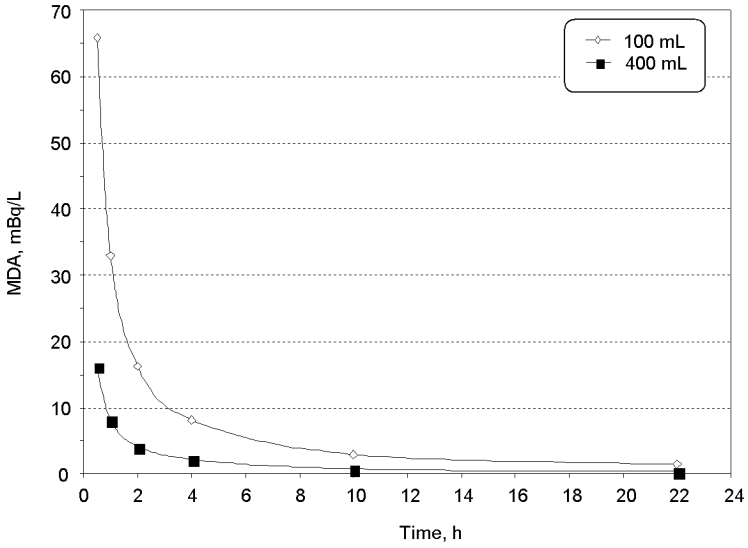

Fig. 3. MDA for actinides vs. time for 100 mL (◊) and 400 mL aliquots (■)

Conclusions

The new method developed in the SRS Environmental Laboratory is a rapid method for the analysis of urine and water samples expected during a radiological emergency response event. This method has high tracer recoveries and effectively removes interferences, such as high levels of ^{210}Po when enhanced column rinsing is employed. Two different options for enhanced ^{210}Po removal were shown to be effective.

The improved report times in the NRIP-2008 Program by the SRS Environmental Laboratory demonstrate the speed and effectiveness of this new method, and illustrate the impact of improvements made such as streamlined calcium phosphate precipitation and much faster column flow rates. For a 100 mL sample aliquot, the MDA for actinides for a 2-hour count time is 16.3 mBq·L^{-1}. For a 400 mL sample aliquot and a 2-hour count time the MDA is 4.07 mBq·L^{-1}. The MDA for ^{90}Sr is calculated in a similar fashion and was determined to be equal to 260 mBq·L^{-1} (7.22 pCi·L^{-1}) for a 400 mL water sample counted for 10 minutes. Longer count times may be used to reduce analytical uncertainty or lower the MDA as needed.

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